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ASSESSMENT OF THE MILITARY APPLICATION OF UPAC, A POLYMERIZED ACRYLAMIDE

R. L. Curtin, et al

Unidynamics/Phoenix, Incorporated Phoenix, Arizona

February 1973

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Disabling internal combustion engines			
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TECHNICAL REPORT NO. LWL-CR-07C71

ASSESSMENT OF THE MILITARY APPLICATION OF UPAC, A POLYMERIZED ACRYLAMIDE

Final Report
Contract No. DAAD05-71-C-0302

Ву

R. L. Curtin Leonard Duran Unidynamics Phoenix, Inc. Phoenix, Arizona

February 1973

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U.S. ARMY LAND WARFARE LABORATORY
ABERDEEN PROVING GROUND, MARYLAND 21005

ABSTRACT

Under contract DAAD05-71-C-0302 with the U.S. Army Land Warfare Laboratory, Unidynamics/Phoenix, Inc. performed a six-month program to evaluate military applications of UPAC, a proprietary acrylamide polymer developed by Unidynamics.

Data characterizing the unique water absorption characteristic of the material was generated, and both laboratory and field feasibility studies were conducted in the following areas:

- Conditioning of tactical surfaces
- Purifying water-contaminated POL products
- Denial of potable water
- Disabling internal combustion engines
- Demonstrating capability of expanding UPAC to perform useful work

It was found that UPAC can be successfully used for all of these applications in varying degrees. It appears to be especially useful for dust suppression, POL decontamination, engine disabling, and performing work, perhaps even a timing function. Denial of standing water supplies is possible, but requires rather large quantities of UPAC. A potential application in this area, however, lies in the use of a water pipe plugging device.

Further studies in all these areas are required for proper development of the concepts involved. LWL's recommended approach to these studies is outlined in the document.

^{*} Irradiated Polymeric Acrylamide

TABLE OF CONTENTS

Section	Page Number
INTRODUCTION	1
MATERIAL PREPARATION	2
CHEMICAL AND PHYSICAL PROPERTIES	3
LABORATORY EVALUATIONS	6
Water Absorption Characteristics	6
Mud Solidification	14
Water Denial	20
Water Removal from Petroleum Products	21
Useful Work	29
Disabling Internal Combustion Engines	33
FIELD TESTING	47
Improvement of Tactical Surfaces	47
Test Area Description	47
Weather Conditions During Test Periods	47
Dust Suppression	47
Mud Solidification	52
Water Removal from POL	53
Disabling Vehicles	54
CONCLUSIONS AND RECOMMENDATIONS	55
Water Removal from POL	55
Useful Work	55
Disabling Internal Combustion Engines	56
Dust Suppression	57

INTRODUCTION

Under the auspices of the U. S. Army Land Warfare Laboratory, Aberdeen Proving Ground, Maryland, Unidynamics/Phoenix began work 5 April 1971 on contract DAAD05-71-C-0302. The objective of the contract was laboratory and field evaluation of the military applications of UPAC, a proprietary acrylamide polymer developed by Unidynamics. The evaluation was to be performed in a period of six months and consisted of:

- Characterization of the water absorbing properties of UPAC over a range of temperature, water hardness and polymer particle size.
- Utilization of the material for conditioning various tactical surfaces, removing dissolved and suspended water from POL products, denial of potable water and disabling internal combustion engines.
- Demonstration of the capability of the material to perform useful work as it absorbed water and expanded.

This document is the final report on the subject contract.

MATERIAL PREPARATION

The preparation of UPAC is accompaished by the following method. A 40 percent by weight solution of acrylamide, $CH_2CHCONH_2$, is prepared by dissolving the monomer in warm distilled water. The solution is stirred until the acrylamide is completely dissolved. Removal of oxygen from the system is important, since oxygen inhibits the formation of the long-chain polymer. The solution is therefore de-aerated in a vacuum for approximately two hours. At this time, the monomer mixture is transferred to a polyethylene bag supported by a metal container. Each bag holds up to four liters of solution. The solution is de-aerated again, this time by passing nitrogen gas through it. The bag is then sealed and is ready for polymerization.

Polymerization is accomplished by exposure to a Cobalt-60 radiation source. The dose is approximately 100 RADs/minute for 100-120 minutes. Initially the polymerized material is firm, rubbery and clear. It is dried at 105°C for five to seven days and then ground to the desired particle size. The dried material is rigid and translucent. In this condition, it is capable of absorbing large quantities of water and growing to many times its dry weight. In water, the UPAC again becomes clear and semi-solid. It retains its original shape on hydration and dehydration.

CHEMICAL ANT: PHYSICAL PROPERTIES

UPAC is a hard, amorphous solid, yellow to off-white in color. On standing in a closed container an ammoniacal odor sometimes develops. The material can be machined easily or ground and bolted to powder of nearly any particle size. The specific gravity of UPAC is 1.25-1.35 g/cc.

The material is a polymer of unknown structure and molecular weight. Ratio of amide/imide bonds is uncertain and probably subject to change with time (suggested by ammonia production on standing). It is insoluble in virtually all solvents studied to date except for liquified phenol (80% phenol in water). The pH of water containing hydrated UPAC is 7.5 to 8.0.

The amount of water absorbed by UPAC and the resultant increase in volume can be varied by changing the acrylamide derivatives which are polymerized. The standard material (a homopolymer of propenoic acid amide) used in the subject studies absorbs 180 times its weight in distilled water.

The presence of ionic solutes in the water to be absorbed, however, depresses both growth rate and (especially) eventual total growth. There has been no effort to characterize this effect in terms of ionic species or concentrations nor has there been a systematic study of the effects of nonionic solutes. Growth in physiological saline (9g NaCl/l) is approximately 15% of growth in distilled water. Growth in "hard" water (520 ppm CaCO₃) is approximately 25 percent of that in distilled water. Variation in temperature between 40° and 100°F has little effect on growth rate and no effect whatsoever on the total growth of UPAC.

When hydrating, the UPAC's tend to retain their shape and generally, the swollen material is identical in configuration with the original. If the swollen material is not deformed in any way

(and most UPAC's are relatively tough in the swollen state) it returns to the approximate original dimensions when dried. This "memory" is a key property in the capability of the material to cycle more or less indefinitely through hydration-dehydration. With some of the special preparations made, recycling was difficult or impossible to achieve because of the soft, gelatinous nature of the hydrated material.

As one would expect from solubility data, fully hydrated UPAC exhibits zero osmotic pressure. While the material is hydrating, however, a considerable head can be developed. When the water and UPAC are separated by a barrier freely permeable to the liquid and impermeable to the polymer, pressures as high as 500-700 psi have been obtained. As indicated in other portions of this document, this pressure can be utilized to do work.

The rate of water absorption is naturally a function of the surface area/weight ratio of UPAC exposed to water. Preliminary data shows that once the surface/mass ratio reaches about 100:1 (mean particle diameter of 0.45 mm or approximately 50 mesh) growth rate reaches a maximum. This maximum is full growth in 15 minutes, provided the growth occurs in an excess of water. At surface-to-mass ratios of 1:1 or less, growth is very slow and may not be attained for several days.

Once water is imbibed in UPAC there is no way of removing it except by drying. Drying may be accomplished between 70°F and 220°F if the ambient relative humidity is 50 percent or less. Drying rate is accelerated, of course, by higher temperatures and is related to surface area/mass. Drying can also be accomplished by Tyophilization procedures but the product of such drying is always a fine powder since freezing the swollen polymer destroys its integrity. Heat and air dried material can be recycled through the hydration step again with no apparent change in absorption capacity. Lyophilized material has not been studied to determine whether or not it, too, can be recycled.

TABLE

		%
Soft H ₂ O	Med. Haid H ₂ O	Hard H ₂ O
58.0	35.5	22.0
98.0	52.5	32.0
123.0	64.0	35.0
145.5	73.0	44.0
•	58.0 98.0 123.0	58.0 35.5 98.0 52.5 123.0 64.0

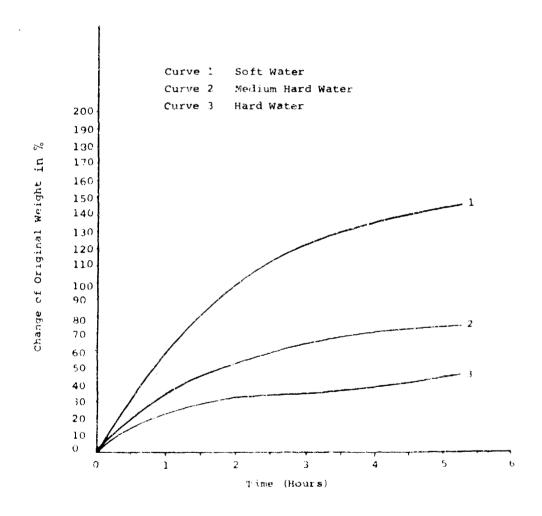


FIGURE 1 COMPARISON OF ABSORPTION RATE $75^{\circ}\text{F} > 10$ MESH SIZE

LABORATORY EVALUATIONS

Water Absorption Characteristics. To determine water absorption characteristics for UPAC, tests were done using three particle sizes (>10, 10-50, 30-60 mesh) at different temperatures and varying hardness of water. The temperatures for the tests were: 40°F, 75°F and 100°F. The grades of water used were: 1) hard-approximately 520 ppm CaCO₃; 2) soft--less than five ppm CaCO₃; 3) medium hard--approximately 260 ppm CaCO₃.

Based on tests performed, water hardness affects the water absorption rate more than the other two factors (temperature, particle size). For a particular particle size and temperature, the water absorption rate of UPAC in soft water is nearly four times as great as that in hard water. (Table I, Figure 1)

Particle size also plays an important part in the water uptake rate of UPAC. The smaller sized particles tested take up water at a very rapid rate, but quickly taper off to a fairly constant level. By comparison, the larger fized particles grow at a slower, more steady rate. After one hour a 30-60 mesh sample grew 212 times its dry weight. However, the material had only grown to 222 times its dry weight after five hours. A greater than 10 mesh sample, under the same conditions, grew only 77.5 times its dry weight after one hour, but had reached 150 times its dry weight after five hours. The smaller sized particles also tend to grow to larger multiples of their initial dry weights than the larger sized particles. For a particular temperature and water hardness, a sample of 30-60 mesh material grew only slightly more than a 10-30 mesh sample, but appreciably more than a sample of >10 mesh material. (Table II. Figure 2)

The temperature of the water used has little effect on the absorption rate. Although warm water appears to facilitate growth, this has not been a universal characteristic. Evidently, influences of

TABLE !! Change of Original Weight in %

Time	Vs	Change of Particle S	ize
(hours)	>10	10-30	30-60
1/2	-	174.0	206.0
1	77.5	196.0	212.0
2	117.0	216.0	212.0
3	131.5	218.0	212.0
5	150.0	221.0	222.0

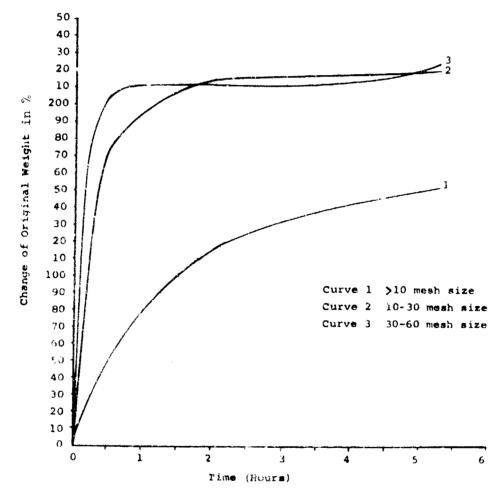


FIGURE 2
COMPARISON OF ABSORPTION RATE AT 100°F IN SOFT WATER

the water quality and particle size overshadow the effect of water temperature. (Table III, Figure 3)

Additional water absorption data is given in Tables IV - VI and Figures 4 - 6.

A summary of the water absorption values of the five different particle sizes of UPAC after four to five hours immersion in water of varying temperatures and degrees of hardness is given in Table VII. Based on these values and certain practical considerations, the 10-30 mesh and 30-60 mesh particle sizes appear to be preferable to the other sizes investigated for most applications. It can be seen from Table VII that UPAC of these two particle sizes is generally equivalent or superior to the other particle sizes in water absorption under a variety of conditions. Although the 60-120 mesh and 120 mesh sizes also have high water absorption rates, these materials are somewhat less desirable to use because they are comparatively difficult to handle. Since both are fine powders, they scatter badly if exposed to any appreciable wind conditions. In addition, the particles tend to clump together when placed in water, even with vigorous stirring or prior wetting with a water-miscible nonsolvent such as acetone. UPAC of >10 mesh particle size may also be less useful in certain applications since it grows at a considerably slower rate than the other sizes and also does not grow to as large a multiple of its original dry weight. This is apparently due to the smaller specific surface area available with the larger particles. The 10-30 mesh and 30-60 mesh particle sizes of UPAC are thus preferred because of their more nearly optimum combination of water absorption and handling characteristics.

TABLE III
Change of Original Weight in %

Time	Ottal	vs Temperature (°F)	
(hours)	40	75	100
1/2	153.0	158.0	174.0
1	182.0	200.0	196.0
2	218.0	202.0	216.0
3	215.0	202.0	218.0
4	213.0	-	-
5	-	202.0	221.0

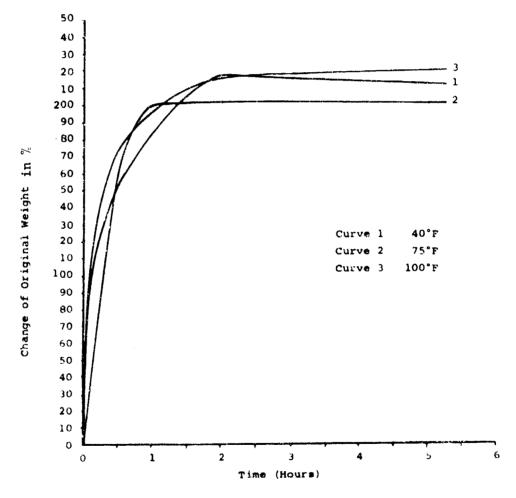


FIGURE 3

COMPARISON OF ABSORPTION RATE OF 10-30 MESH SIZE IN SOFT WATER

Water Absorption Rate in Soft, Medium & Hard Water

Time	Mult	iple of Original Weig	ht
(hours)	Soft H ₂ O	Med. Hard H ₂ O	Hard H ₂ O
1/4	200	75	50
1/2	200	75	50
1	200	75	50
2	200	75	50
3	200	75	50
4	200	75	50

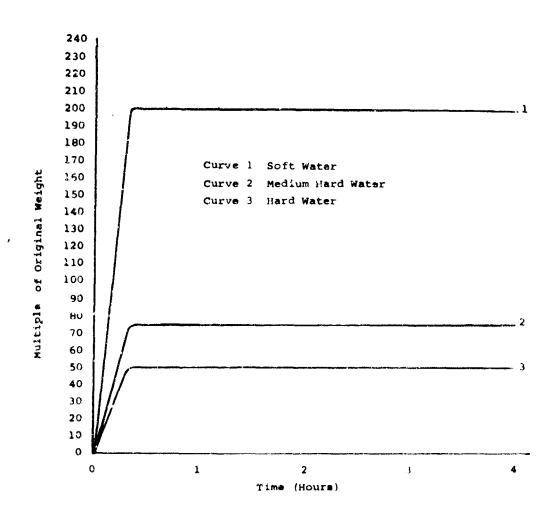


FIGURE 4

COMPARISON OF WATER ABSORPTION RATES OF UPAC AT 100 F,

<120 MESH PARTICLE SIZE

TABLE V
Water Absorption Rate vs Temperature

Time	Multiple of Original Weight				
(hours)	40°F	75°F	<u>100</u> °F		
1/4	50	48	55		
1/2	50	48	55		
1	50	48	55		
2	50	48	55		
3	50	48	55		
4	50	48	55		
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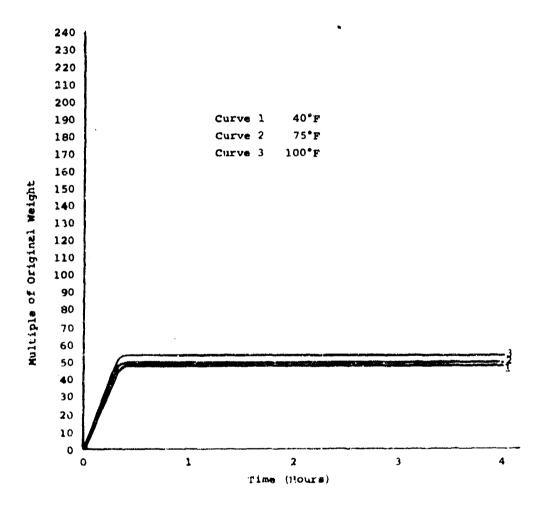


FIGURE 5
COMPARISON OF WATER ABSORPTION RATES OF 60-120 MESH
UPAC IN HARD WATER

TABLE VI
Water Absorption Rate vs Particle Size

Time	Multiple of Original Weight					
(hours)	>16	10-30	30-60	69-120	<120	
1/4	-	_	<u></u>	205	200	
1/2	-	153	216	205	200	
1	39	182	226	205	200	
2	73	218	226	205	200	
3	101	215	226	205	200	
4	125	213	226	205	200	

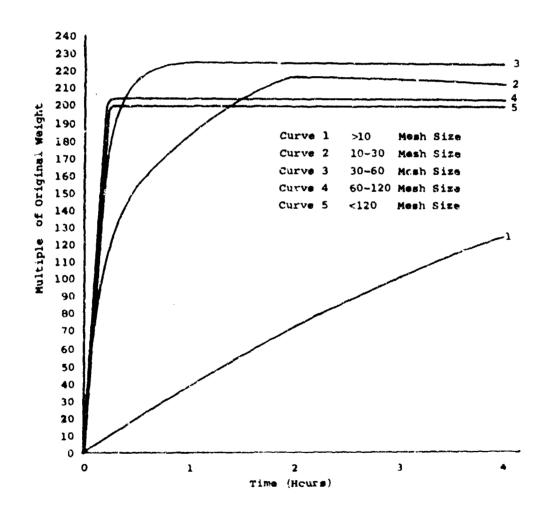


FIGURE 6

COMPARISON CF ABSORPTION RATES OF DIFFERENT UPAC
PARTICLE SIZES AT 40°F IN SOFT WATER

TABLE VII

COMPARISON OF WATER ABSORPTION VALUES OF DIFFERENT PARTICLE SIZES OF UPAC AFTER 4-5 HOURS UNDER VARYING CONDITIONS OF WATER TEMPERATURE AND HARDNESS

Water		Size of	Particle		
Conditions	>10	10-30	<u> 30-60</u>	60-120	<120
soft H ₂ O, 40°F	140.0	213.0	228.0	205.0	200.0
Medium Hard H ₂ O, 40°F	60.5	88.0	82.0	0.08	75.0
Hard H ₂ O, 40°F	42.0	60.0	50.0	50.0	50.0
Soft H ₂ O, 75°F	145.5	202.0	220.0	200.0	195.0
Medium Hard H ₂ O, 75°F	73.0	74.0	70.0	80.0	75.0
Hard H ₂ O, 75°F	44.0	56.0	52.0	48.0	50.0
soft н ₂ 0, 100°F	150.0	218.0	222.0	200.0	200.
Medium Hard H ₂ O, 100°F	74.5	75.0	76.0	75.0	75.
Hard H ₂ O, 100°F	50.5	54.0	50.0	55.0	50.

Mud Solidification. Tests to determine the effectiveness of UPAC as a mud solidification agent were conducted. For these tests, local soil was screened through a No. 10 sieve to remove any foreign materials and to assure sample homogeneity. Initially, the screened soil was oven-dried, but this operation was found to be unnecessary and was subsequently eliminated. The tests were conducted using two-inch thick, four-inch thick, and sixinch thick soil samples containing either 20 percent or 30 percent by weight of soft (distilled) water. All five particle sizes of UPAC were evaluated to determine their relative abilities to firm up the soil. Initially, the UPAC was merely distributed over the surfaces of the wet soil samples, but it was found that mixing the material into the soil was highly advantageous in practically all cases and essential in some. For this reason, the UPAC was mixed into all of the six-inch deep samples and some of the twoinch thick and four-inch thick samples. Three ratios of UPAC to water were employed in the initial tests. These were 1:50, 1:75 and 1:100. Later, only the 1:100 ratio was used, since the other two ratios, requiring more UPAC, did not appear to increase the effectiveness of the operation.

The apparatus used in the soil conditioning tests consisted of a weighted vertical rod assembly that imparted a pressure of 25 pounds per square inch when rested on the soil sample. The rod, supported by a collar and a base, was attached to a 0.2194-inch diameter bolt at the lower end where it contacted the soil sample, and contained a platform for attaching weights at the top. The weight of the rod and its associated items was counterbalanced to allow the bolt to be free-standing. Under these conditions, a total weight of 429 grams was required on the 0.0378-square inch surface area of the bolt to equal the desired pressure of 25 pounds per square inch.

The overall objective of the series of experiments was to correlate the quantity and particle size of UPAC with the time required to attain a surface yield strength of 25 pounds per square inch in the

soil. In most cases the tests were terminated after 24 hours if the desired yield strength had not been achieved. The results of these tests are presented in Tables VIII, IX and X, which reflect data for soil samples of two inches, four inches and six inches depth, respectively.

Based on the laboratory scale soil conditioning experiments, several conclusions can be drawn concerning the best methods and materials to use in the field assessment tests. First, it is apparent from the laboratory studies that the UPAC granules should be mixed into the upper portion of the soil. Otherwise, as the UPAC absorbs the surface water and expands, it swells in an upward direction away from the level of the soil. Extremely thorough mixing is probably not required. However, it is recommended that the UPAC be blended into at least the upper two to four inches of soil.

In addition to the effect of mixing, a second major factor in soil conditioning is the particle size of the UPAC employed. on the experiments conducted to date, it appears that moderately coarse granules (10-30 mesh) of UPAC result in a higher surface yield strength in the soil than finely ground material. From a time efficiency standpoint, the best particle size of UPAC to use in soil conditioning appears to be 30-60 mesh. In the six-inch deep soil samples, the use of this material resulted in a yield strength of nine pounds per square inch within 45 minutes and 25 pounds per equare inch in 22 hours. The use of UPAC in the 10-30 mesh and >10 mesh particle size ranges produced essentially the same yield strength after 24 hours as the 30-60 mesh material. However, the short term efficiency of the coarser sizes was somewhat inferior to the 30-60 mesh product. If several hours can be allowed for conditioning the soil prior to its use as a support surface, it should make little or no difference whether UPAC of >10 mesh, 10-30 mash or 30-60 mesh is employed. In this case, it would be advantageous to use the >10 mesh material, since it would be less expensive than the finer grinds. However, if very rapid conditioning of the soil is a major consideration, the use of 30-60 mesh UPAC is strongly recommended,

RESULTS OF SOIL CONDITIONING TESTS USING TWO-INCH-DEEP SOIL SAMPLES

TABLE VIII

Test No.	UPAC Particle <u>Size</u>	Water Content in Soil (%)	UPAC: Water Ratio	Soil Surface Yield Strength Versus Time
1	>10	20	1:75	3-1/4 hours - 25 psi
2	>10	20	1:100	3-1/2 hours - 25 psi
3	>10	30	1:75	Did not attain 25 psi in 24 hrs.
4	>10	30	1:100	Did not attain 25 psi in 24 hrs.
5	10-30	20	1:75	3-1/2 hours - 25 psi
6	10-30	20	1:100	3-1/2 hours - 25 psi
7	10-30	30	1:75	Did not attain 25 psi in 24 hrs.
8	10-30	30	1:50	~ 24 hours - 25 psi
9	30-60	20	1:75	3 hours - 25 psi
10	30-60	20	1:100	3-1/4 hours - 25 psi
1.1	30-60	30	1:50	Did not attain 25 psi in 24 hrs.
12	30-60	30	1:75	Did not attain 25 psi in 24 hrs.
13	60-120	20	1:100	1/2 hour - 17 psi 1-1/4 hours - 25 psi
14	60-120	30	1:100 ⁽¹⁾	1/4 hour - 17 psi 1/2 hour - 18 psi 1 hour - 22 psi 1-1/2 hours - 25 psi
15	<120	20	1:100(1)	3/4 hour - 25 psi
16	<120	30	1:100 ⁽¹⁾	~24 hours - 25 psi

⁽¹⁾ UPAC mixed into the soil sample

TABLE IX

RESULTS OF SOIL CONDITIONING TESTS USING FOUR-INCH-DEEP SOIL SAMPLES

Test.	UPAC Particle Size	Water Content in Soil (%)	UPAC: Water <u>Ratio</u>	Soil Surface Yield Strength Versus Time
1	>10	20	1:100	3 hours - 25 psi
2	>10	30	1:100	2-1/2 hours - 5 psi 4 hours - 8 psi 22-1/2 hours - 20 psi ~24 hours - 25 psi
3	10-30	20	1:100	1/2 hour - 18 psi 1-1/2 hours - 25 psi
4	10-30	30	1:100	1/2 hour - 5 psi 1-1/2 hours - 7 psi 3 hours - 9 psi 22 hours - 14 psi 40 hours - 25 psi
5	30-60	20	1:100	3/4 hour - 16 psi 1 hour - 19 psi 1-3/4 hours - 25 psi
6	30-60	30	1:100	3/4 hour - 4 psi 1 hour - 5 psi 1-3/4 hours - 9 psi 6 hours - 11 psi (1)
7	60-120	20	1:100	3/4 hour - 17 psi 2 hours - 25 psi
8	60 -12 0	30	1:100	3/4 hour - 6 psi 2 hours - 10 psi 5 hours - 12 psi 6-1/2 hours - 16 psi 24 hours - 25 psi
9	<120	20	1:100	1 hour - 16 psi 2 hours - 22 psi 2-1/2 hours - 25 psi
10	<120	30	1:100	Did not attain 25 psi in 24 hours (2)

⁽¹⁾ Test discontinued after six hours

⁽²⁾ Surface glaze formed which prevented the water underneath from being absorbed by the UPAC

TABLE X

RESULTS OF SOIL CONDITIONING TESTS USING SIX-INCH-DEEP SOIL SAMPLES

Test No.	UPAC Particle Size	Water Content in soil (%)	UPAC: Water <u>&atio</u>	Soil Surface Yield Strength Versus Time
0	> ∙10	30	1:100 (1)	3/4 hour - 5 psi hour - 10 psi 2 hours - 13 psi 5 hours - 17 psi 6 hours - 18 psi 22 hours - 23 psi
2	10-3	30	1:100(1)	3/4 hour - 7 psi 1 hour - 10 psi 2 hours - 12 psi 4-1/2 hours - 15 psi ~24 hours - 25 psi
3	30-60	30	1:100 (1)	3/4 hour - 9 psi hour - 11 psi 2 hours - 13 psi 4 hours - 17 psi 22 hours - 25 psi
4	60-120	30	1:100 (1)	3/4 hour 10 psi 1 hour - 12 psi 2 hours - 15 psi 3 hours - 16 psi 5 hours - 20 psi 6 hours - 20 psi 24 hours - 20 psi
Ē	<120	30	1,100 (1)	3/4 hour - 5 psi hour - 5 psi hour - 7 psi hours - 7 psi hours - 9 psi hours - 9 psi hours - 9 psi hours - 17 psi hours - 17 psi

⁽¹⁾ UPAC mixed into the soil sample

<u>Water Denial</u>. The capacity of UPAC to absorb and hold large quantities of water suggested that the material could be used to deny water supplies to hostile forces in arid and semi-arid locales. For purposes of the preliminary evaluation of this use it was presumed that two kinds of water supplies would be involved: standing pools (tanks, catch basins, ponds, reservoirs, etc.) and enclosed (well casings, piping, etc.).

Calculation showed that complete denial of standing supplies would require considerable quantities of UPAC. In hard water approximately 54,400 pounds of the polymer would be needed to completely absorb one acre-foot of water. In soft water that amount would fall to 13,700 pounds/acre-foot. For smaller quantities of free standing water UPAC requirements for denial are 170 pounds/1,000 gallons (hard) and 43 pounds/1,000 gallons (soft). It should be noted, however, that potable water treated in this manner is permanently denied since absorbed water recovered from UPAC by conventional methods (heat, vacuum distillation, lyophilization, etc.) is contaminated with ammonia.

A design for a device was developed to demonstrate water denial in an enclosed surply. This device consists of a perforated cylindrical container supporting a sample of UPAC in a manner which will allow it to expand only in a radial direction. See Figure 7. The advantages of such a device are that the center and ends are made of metal (aluminum, steel, etc.) and would be difficult to rupture.

The ability of the device to restrict water flow was excellent. After allowing the UPAC to grow, water flow was totally restricted. However, friction against the walls of the pipe was low and the device could be easily moved along the tube. We recommend either employing + device with a sheath having high friction or putting the device as presently designed where it will not move (joint, elbow, etc.).

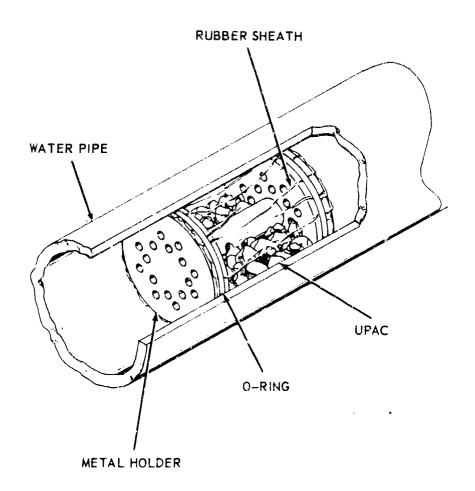


FIGURE 7
RESTRICTION DEVICE

Another restriction device was designed using a cloth bag. UPAC was placed in the bag which was sealed and inserted in a pipe with water. The UPAC (>10 mesh) grew well and restricted water flow. However, the seams had a tendency to rip and the cloth material is subject to puncture and easy removal. The device does, however, restrict water flow effectively and the friction between wall and fabric is high (76.4 psi).

Ail restriction devices used >10 mesh UPAC. Cloth bags used 10 grams of UPAC, while the metal restriction device was loaded with five grams.

Water Removal from Petroleum Products. Although having a great capacity for scavenging water, UPAC is inert in the presence of petroleum products. It therefore seemed likely the material could be used to remove water contaminants from POL.

To evaluate this application UPAC was used to remove water from JP-4 jet fuel, 115-145 aviation fuel, regular grade commercial gasoline, diesel oil, kerosene and lubricating oil. Three techniques were used for water removal: (1) The UPAC was added to fuel samples as loose powder, (2) cloth bags containing UPAC were immersed in fuel samples, and (3) the fuel was passed through loosely packed columns of UPAC. Water content of treated and untreated samples of fuel was determined by the Karl Fisher Method (ASTM-D1744-64). Treated fuels were studied to determine whether or not they were contaminated with UPAC by exposure to that material.

In preliminary tests, one milliliter of distilled water was added to 99 milliliters of petroleum product and the fuel shaken vigorously to disperse the contaminant. One gram of 10-30 mesh loose granular UPAC was added to the wet POL sample and the mixture allowed to stand. At the end of one-hour, two-hour and 24-hour intervals the UPAC was collected on a filter and weighed. The results of these tests are shown in Table XI.

TABLE XI

WEIGHT INCREASE OF 10-30 MESH UPAC VERSUS TIME OF CONTACT WITH WATER-CONTAMINATED POL PRODUCTS

	UPAC Weight (grams)				
Contact Time (hours)	JP-4 <u>Jet Fuel</u>	115/145 Aviation Fuel	Lubricating Oil	Regular Gasoline	
0	1.00	1.00	1.00	1.00	
1	2.00	2.02	1.76	2,23	
2	2.02	2.03	1.84	2.30	
24	2.00	2,00	1,85	2.25	

 $1 \text{ ml H}_20 = 1 \text{ grsm}$

The tabulated weight gains in UPAC in contact with "wet" MP-4, aviation fuel and regular gasoline clearly show that the contaminating wat r was absorbed by the polymer in a period of an hour. The excess weight gained by the sample exposed to regular gasoline was apparently not due to absorption of the fuel itself since it held constant with time. Tentatively, the excess was ascribed to selective absorption of water soluble additives in the fuel. Poor results observed with lubricating oil were ascribed to the high viscosity of that material.

Additional samples of JP-4, aviation fuel, lubricating oil and regular grade gasoline were contaminated with one ppm water. One gram of 10-30 mesh UPAC was added as loose powder to the contaminated sample. After 24 hours the POL materials were decanted from the UPAC and analyzed for water content by the Karl Fisher method already referenced. The results are tabulated in Table XII.

TABLE XII

WATER CONTENT OF POL SAMPLES BEFORE AND AFTER TREATMENT
WITH LOOSE UPAC

	Water Content (ppm)		
	As Received (1)	After Contamination (2)	After Treatment
JP-4	40.2	10 ⁴	50.3
115/145 Aviation Fuel	34.7	104	40.1
Regular Gasoline	68.1	104	54.4
Lubricating Oil	17.6	104	130.6
Kerosene	79.6	104	35.0

⁽¹⁾ The "normal" concentration of water in the material before purposeful contamination by Unidynamics.

Columns were prepared by adding 20g of loosely packed, 10-30 mesh UPAC to standard 25 mm diameter chromatographic funnels with medium porosity glass frit. The columns were approximately two inches in height. POL samples were passed through such columns by gravity flow before and after addition of one pph water. The effluents from the column were analyzed for water content as already described. The results are tabulated in Table XIII below:

TABLE XIII

MOISTURE CONTENT OF POL SAMPLES BEFORE AND AFTER TREATMENT IN UPAC COLUMNS

	Moisture Content (ppm)			
	As Received Material		Contaminated Material	
	Before Column	After Column	Before (1)	Arter Column
JP-4	40.2	19.5	104	20.7
115/145 Aviation Fuel	34.7	16.4	104	13.4
Regular Gasoline	68.1	15.3	104	38.6
Lubricating Oil	17.6	22.1	104	172.5

⁽¹⁾ Estimated by calculation.

⁽²⁾ Estimated by calculation.

Further study of column effectiveness was performed with the equipment setup shown diagrammatically in Figure 8. The columns used were 0.44 inch in diameter. They were charged with three or six grams of 10-30 mesh UPAC which resulted in a column length of two inches or four inches respectively (bulk density of 10-30 mesh UPAC approximately 38 pounds/cubic feet). Flow through the columns was controlled by regulating the head pressure or changing the diameter of the exit crifice.

Kerosene was chosen as a representative of material. About 20 gallons of that liquid was contaminated with approximately one part per thousand water and allowed to equilibrate overnight after thorough mixing. At each different flow rate studied, approximately four liters of kerosene were passed through the column. The last liter was sampled and the water content determined by the Karl Fisher method. A fresh column was employed at each flow rate. The results obtained are tabulated in Table XIV below and shown graphically in Figure 9.

MOISTURE CONTENT OF KEROSENE TREATED IN UPAC COLUMNS

3g Column		6g Column			
Flow R	ate(1)	Effluent Moisture Content	Flor	w Rate	Effluent Moisture Content
Liters/min.	Gallons/min(1)	(ppm)	1pm	<pre>gpm(2)</pre>	(ppm)
.14	7	20.5	.26	13	24.0
.74	36	53.9	.66	32	46.3
1.04	51	57.2	.80	39	49.6
1.44	71	59.2	.98	48	58.3

⁽¹⁾ Calculated value on basis of increasing column diameter to six inches and column charge to 1.2 pounds.

⁽²⁾ Calculated value as above with column charge of 2.4 pounds.

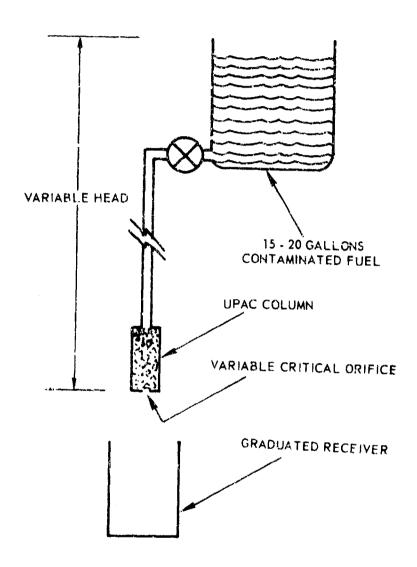


FIGURE 8

APPARATUS FOR STUDY OF DEWATERING POL LIQUIDS
WITH UPAC COLUMNS

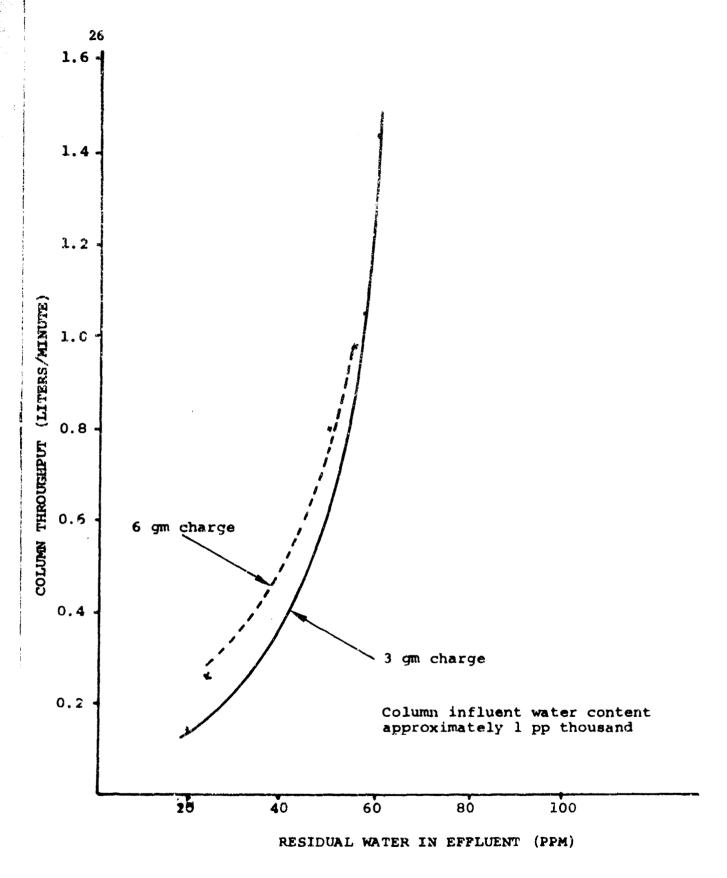


FIGURE 9
REMOVAL OF WATER FROM KEROSENE WITH UPAC COLUMNS

A series of tests were performed to determine (1) whether or not UPAC absorbed the POL liquids under test, and (2) whether or not the POL liquids had any solvent action on the polymer over prolonged period of contact.

Large solid sections of UPAC were carefully weighed and then immersed in the various test liquids for 72 hours. After that period, the UPAC was removed from the liquid, blotted dry and reweighed. The results below clearly show that none of the POL materials are absorbed.

TABLE XV
72-HOUR POL ABSORPTION TEST RESULTS

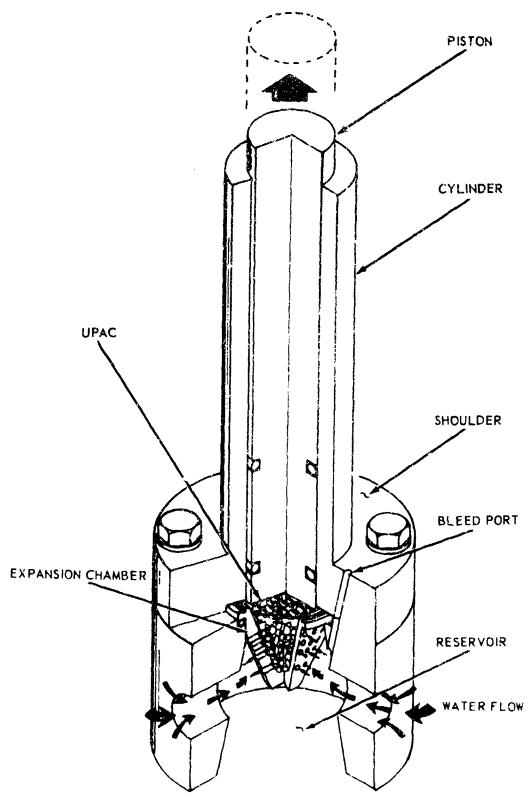
POL	Original Weight	Weight of 72 Hour Immersion (g)
JP-4	10.6325	10.6315
115/145 Aviation Gas	10.3010	10,2984
Regular Gas	9.7527	9.7506
Lubricating Oil	7.1648	7.1650

Measured volumes (10.00 ml) of POL liquids were kept in contact with one g samples of 10-30 mesh UPAC for one week at a temperature of 75 F. After that period the UPAC was removed by filtration and the filtrate evaporated to dryness on a steam bath along with control samples of the test liquids. The results tabulated below show that no UPAC was dissolved over the interval studied.

TABLE XVI

ONE-WEEK POL SOLVENT ACTION TEST RESULTS

	Non-Volatile Solids (q)		
	Control	In Contact with UPAC	
JP-4	0.0002	0.0014	
	0.0012	0.0003	
115/145 Aviation Gas	0.0005	0.0003	
	0.0027	0.0009	
Regular Gasoline	0.0005	0.0002	
	0.0103	0.0109	



Scale: Approximately 3-1 of Actual Size

FIGURE 10
USEFUL WORK DEVICE

Useful Work. It was known that relatively high pressures can be generated when UPAC is confined as it absorbs water. suggested that the expansion of UPAC could be harnessed to produce useful work. As part of the subject effort, a device was designed for demonstrating this capability of UPAC. Figure 10 is a sketch of the device. Its operation is quite simple. The conical expansion chamber is lined first with a fine screen and then with analytical grade filter paper (120 mesh screen and Whatman No. 1 paper are adequate). The lined chamber is then filled level with large particles (greater than 10 mesh) of UPAC. The piston is removed, the cylinder is bolted in place, and the assembly immersed in water just deep enough to cover the shoulder. After inspection of the interior of the expansion chamber to make sure the UPAC is thoroughly wet, the piston is inserted and bottomed on the surface of the polymer; the air forced from the cylinder is vented cut of the expansion chamber and reservoir through a bleed port located in the body of the device. As the UPAC expands it is prevented from extruding through the perforations in the chamber by the screen reinforced fiber mat of the filter paper. The expanding UPAC thus forces the piston to rise.

Data taken with the subject device are plotted in Figures11 and 12. Figure 11 shows the distance traveled by the piston under constant load conditions. Replicate data points obtained are indicated along each curve. The dashed portion of the 60.5 pound load line was extrapolated using a smooth curve. Figure 12 shows the same data in a semi-logarithmic plot with load given in psi rather than dead weight and piston travel given as average rate (calculated by dividing travel in inches by time in hours).

Study of the latter figure indicates that the upper limit of load with which useful work may be obtained by expansion of UPAC is approximately 700 psi. This value is apparently the maximum back pressure against which UPAC will absorb water.

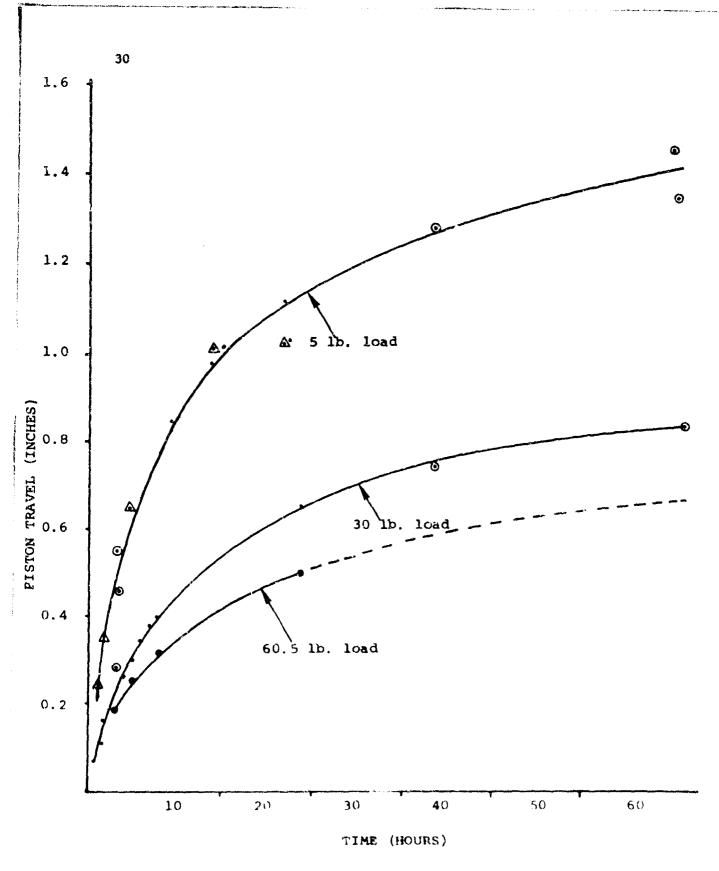
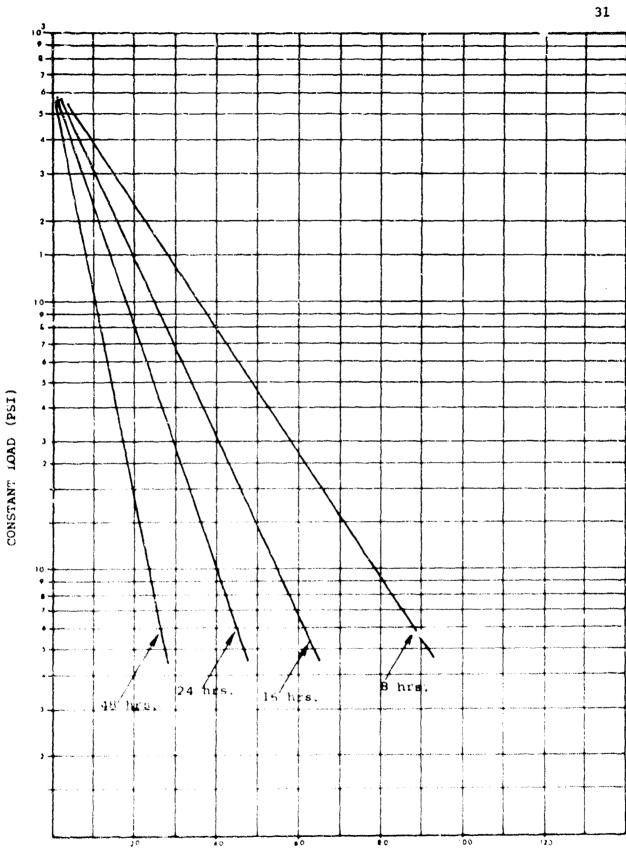


FIGURE 11
PISTON TRAVEL UNDER CONSTANT LOAD



RATE OF PISTON TRAVEL UNDER VARIOUS LOAD CONDITIONS

RATE OF PISTON TRAVEL (.001"/HR)

FIGURE 13

PISTON TRAVEL IN USEFUL WORK DEVICE

UNDER FOUR FOUR SEPARATE GROWTH CYCLES

The precision of the useful work device was evaluated by measuring piston travel over four separate cycles of approximately 10 days duration. The results of those measurements are shown graphically in Figure 13.

Disabling Internal Combustion Engines. In the laboratory phase an electric generator set was used to ascertain the usefulness of UPAC in disabling cooling and fuel systems of internal combustion engines. The generator produces 13 amperes and 1.5 KVA, and is powered by a Krohler Model No. 1E21H four-cylinder engine with an output of four horse power at 1,200 rpm. The coolant used was a 50/50 mixture of commercial glycol antifreeze and water. Engine temperature was monitored by installing thermocouples as shown in the diagram in Figure 14. Figures 15 and 16 show the temperatures reached in the engine under normal operation conditions of no load and full load, respectively.

First efforts (Figures 17 - 19) were unsuccessful in terms of overheating the engine. Internal inspection of the cooling system indicated that the failure was probably due to the inability of the large particle size material to effectively block water passages and galleries.

Further tests were conducted using UPAC in finer mesh sizes. The results (Figures 20 - 23) clearly showed that 50 gram charges of either 10-30 or 30-60 mesh UPAC effectively incapacitated the cooling system. They further showed that 30-60 mesh material was superior in this regard.

Additional tests (Figures 24 and 25) showed that rapid ircapacitation of the engine was possible with even 30 gram charges of 30-60 mesh UPAC.

After each of the tests described above a major effort was required

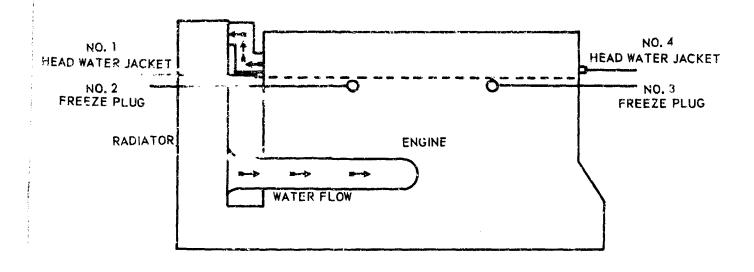


FIGURE 14
THERMOCOUPLE PLACEMENT ON TEST ENGINE

to clear the cooling system of swollen UPAC. It was necessary to remove and flush the radiator, hoses and the head. The cooling galleries and passages in the block were cleared by removal of the four freeze plugs and back-flushing with water. It was clear on inspection of the condition of the cooling system that the effort described was necessary if further operation of the engine was desired. To continue operation would have resulted in serious internal damage due to heat buildup.

Text continued on Page 47.



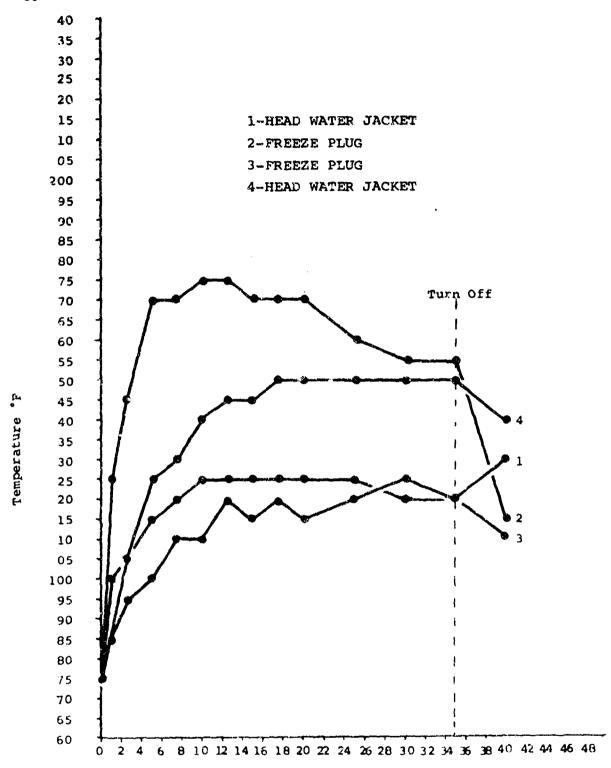


FIGURE 15

HORMAL OPERATING CONDITIONS
HO LOAD

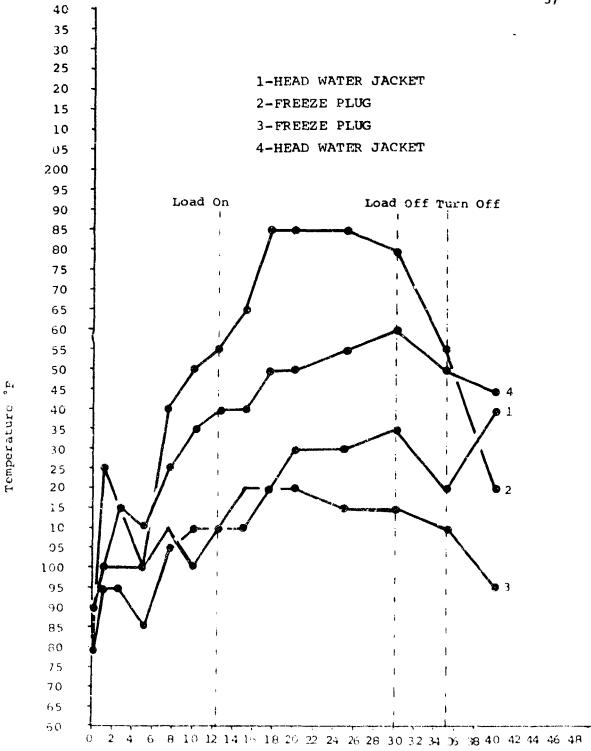


FIGURE 16

NORMAL OPERATING CONDITIONS
WITH LOAD



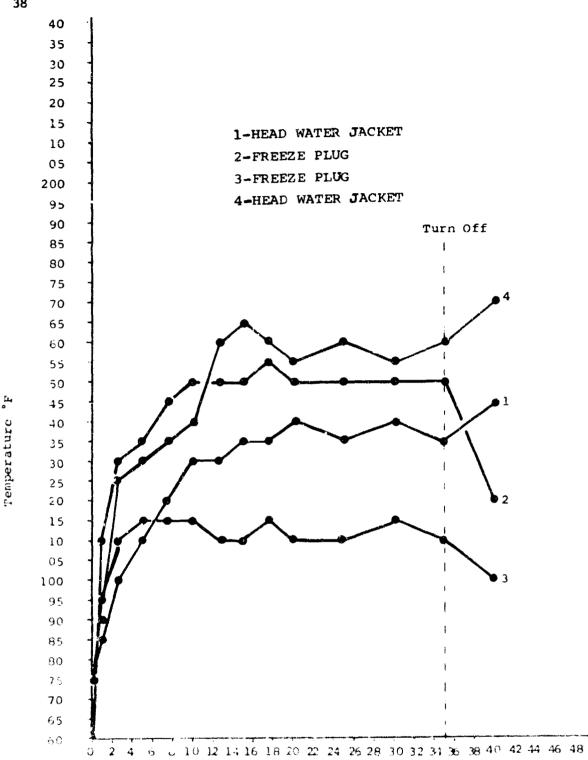
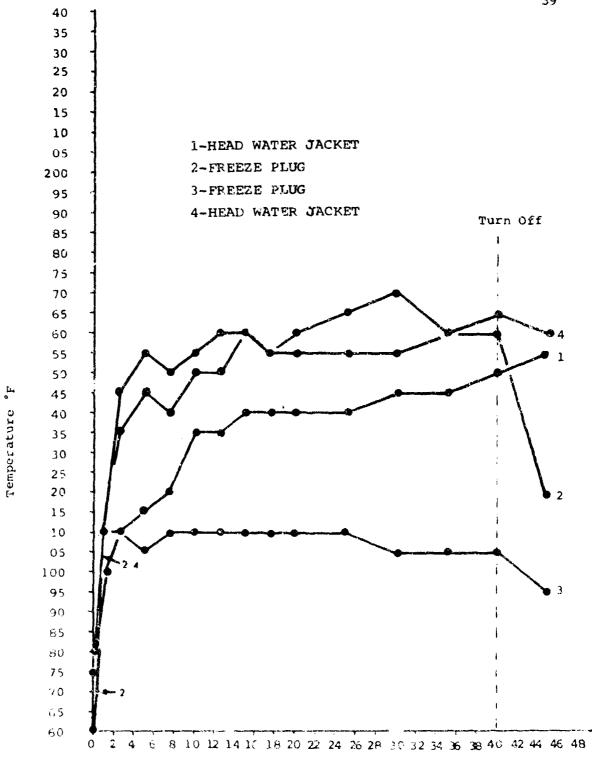


FIGURE 17

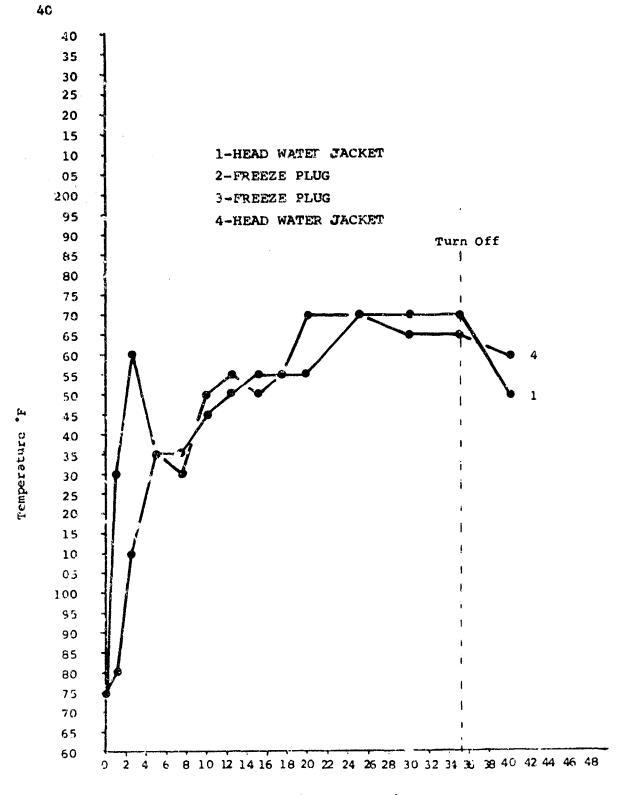
ENGINE DISABLE TEST NO. 1 20 GM UPAC >10 MESH, IMMEDIATE STAPT, WITH LOAD



Time (Minutes)

FIGURE 18

E TIME DISABLE TEST NO. 3 20 GM UPAC >10 MESH, FIVE-HOUR WAIT BEFORE START, WITH LOAD



Time (Minutes)

FIGURE 19

ENGINE DISABLE TEST NO. 3
50 GM UPAC >10 MESH, FIVE-HOUR WAIT BEFORE START, WITH LOAD



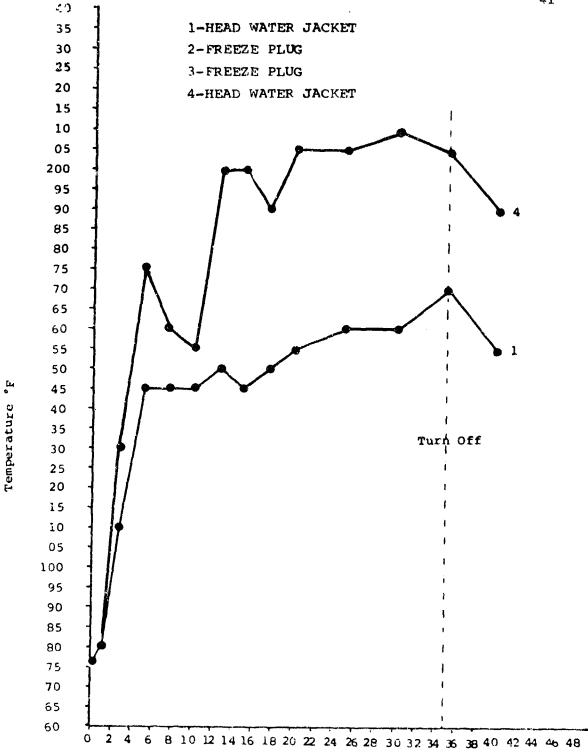
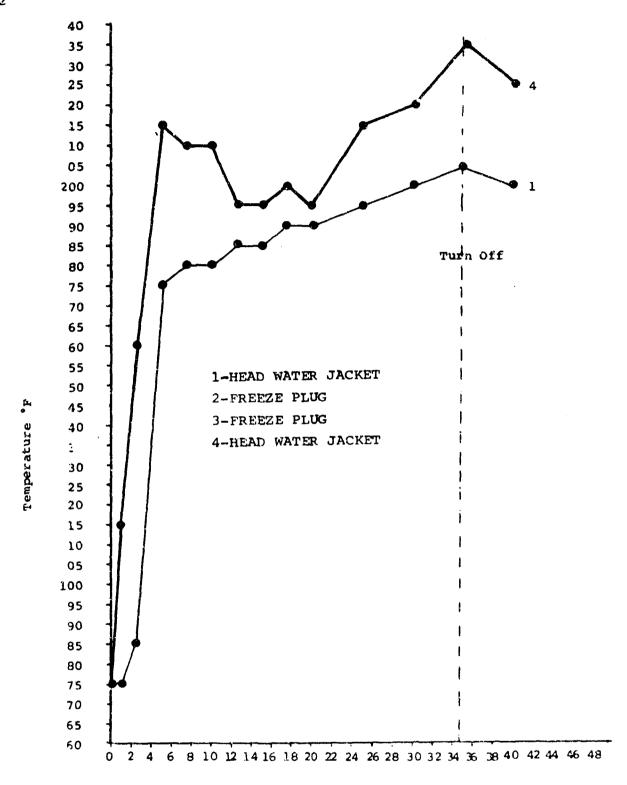


FIGURE 20

ENGINE DISABLE TEST NO. 4
50 GM UPAC 10-30 MESH, IMMEDIATE START, WITH LOAD



Time (Minutes)

FIGURE 21

ENGINE DISABLE TEST NO. 5

50 GM UPAC 10-30 MESH, FIVE-HOUR WAIT BEFORE STARY, WITH LOAD



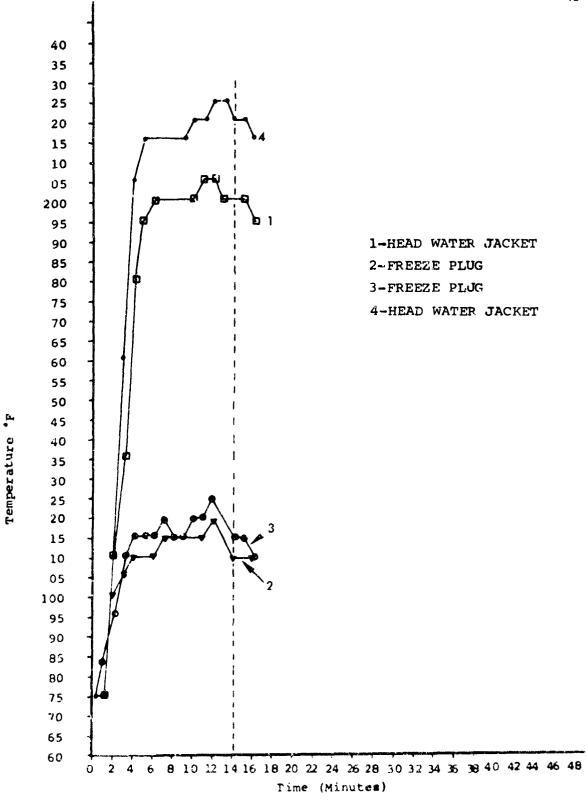


FIGURE 22

ENGINE DISABLE TEST NO. 6

50 GM UPAC 30-60 MESH, IMMEDIATE START, WITH LOAD



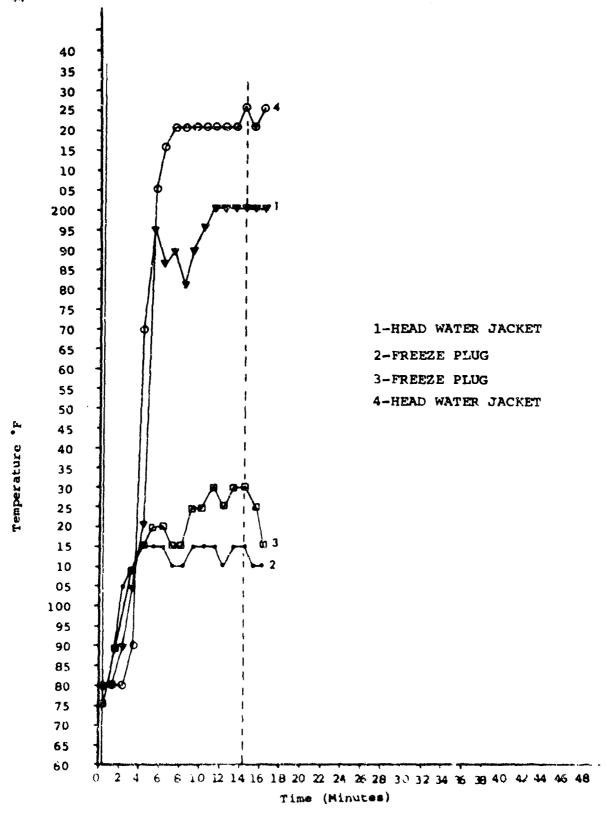


FIGURE 23

SNGIHE DISABLE TEST NO. 7

50 CM UPAC 30-60 MESH, FIVE-HOUR WAIT REFORE STARY

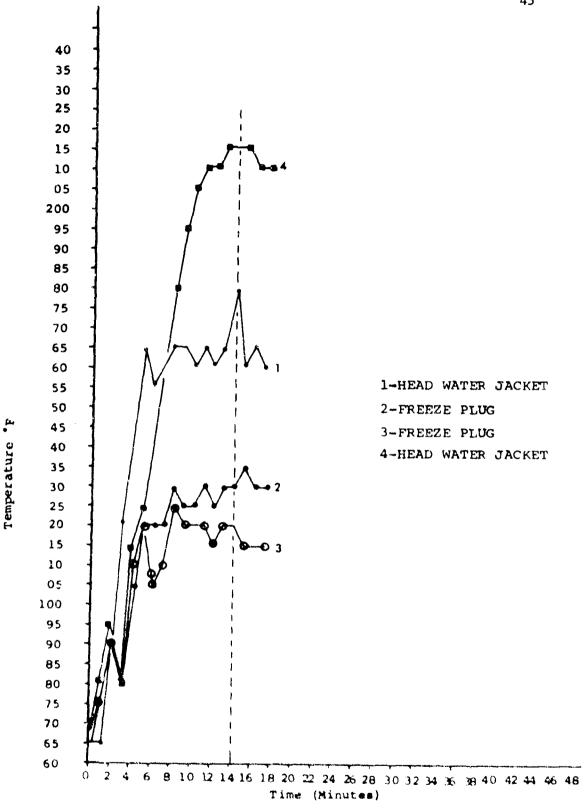


FIGURE 24

ENGINE DISABLE TEST NO. 8

30 GM UPAC 30-60 MESH, IMMEDIATE START



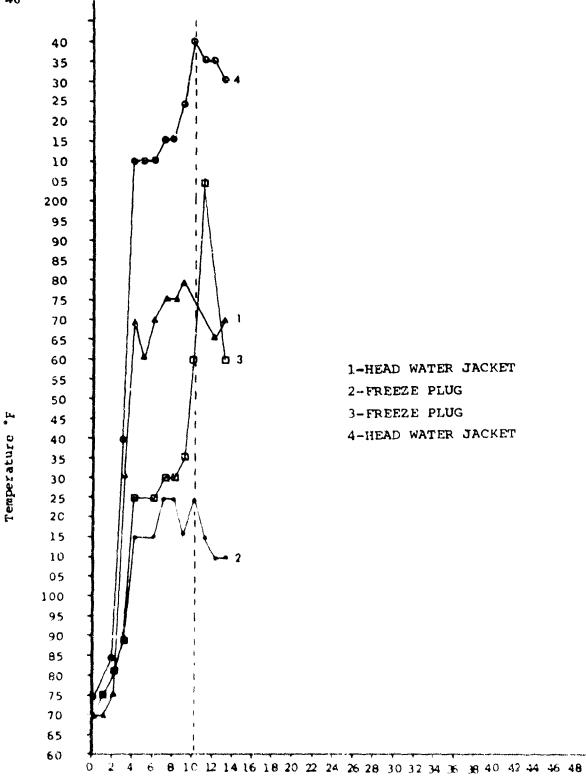


FIGURE 25

ENGINE DISABLE TEST NO. 9 30 GM UPAC 30-40 MESH, FIVE-HOUR WAIT BEFORE START

FIELD TESTING

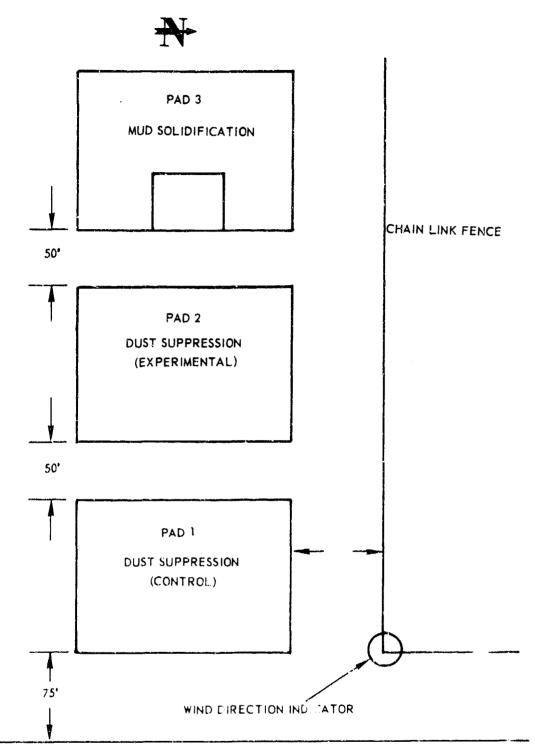
Improvement of Tactical Surfaces. Objective: UPAC was tested as a dust suppressant on helicopter landing pads and as a means of improving the load bearing quality of mud. The tests were performed at Unidynamics' plant site using a UH-IH sircraft provided by the government. Personnel from Land Warfare Laboratory were on hand to observe these tests.

Test Area Description - Three 75 foot square pads were prepared on Unidynamics' plant site. The pads were arranged as shown in Figure 26. The ground on which the pads were located was the dry, sandy clay typical of the southwest desert. Because there was no ground cover and there had been no significant rainfall in over two months, the test area was exceptionally dusty.

weather Conditions During Test Periods - Figure 27 graphically shows air temperature and humidity during the three-day test period. The data was obtained from the operational weather squadron at Luke AFB which is situated approximately eight miles due north of Unidynamics' plant site. Except for a brief period of high wind (30-50 mph out of the northwest at approximately 1800 hours on the evening of September 15) the weather was clear at all times. Prevailing winds were out of the scutheast in the morning and the southwest in the atternoon. Prevailing wind velocity ranged from dead calm to about five mph.

Surface temperatures on the test pads were not measured. Unidynamics' experience indicates, however, that values as high as 125-135°F are typical in the kind of weather experienced during the test period.

Dust Suppression - At approximate, y 0830 hours on 14 September 1971, 40 pounds of 10-30 mesh UPAC were evenly distributed on Pad 2, using hand carried spreaders normally employed for seeding lawns. After



PARKING LOT

FIGURE 26
TACTICAL SURFACE IMPROVEMENT TEST AREA

spreading was complete (15 minutes, two men) the surface of the pad was rolled with a leveling roller suspended from a three-point hitch on a Ford Ferguson Tractor. Immediately after rolling was complete, Pads 1 and 2 were sprinkled with a total of about 5,000 gallons of water. The pads were then left to "cure" until testing began four hours later.

The results of testing are described below and fully documented in 35 mm color slides and 16 mm color move footage

14 September 1971 - Both the control and treated pads were still somewhat damp when testing began. The high moisture content of the soil and the surface crust formed by sprinkling combined to create low dust from both pads when the test aircraft hovered and/or approached. Considerable dust was raised from the surrounding area, however, and it became clear that it would be necessary to thoroughly wet these areas in order to evaluate test results properly. During this first day of testing the aircraft hovered and/or approached the test and control pads for a period of about two to three hours.

15 September 1971 - Testing was resumed at 1000 hours, approximately 26 hours after test pad preparation. Just prior to arrival of the test aircraft the areas surrounding the test pads were dampened thoroughly. No water was added to the experimental or control pad surfaces. The crust of these surfaces was broken up by heavy crisscross traffic of two Ford Ferguson tractors.

Inspection of the pad surfaces showed that the control area was quite dry and powdery. The surface treated with UPAC, however, was still somewhat moist and had a "tacky" feel.

Temperature

- - - - - Humidity

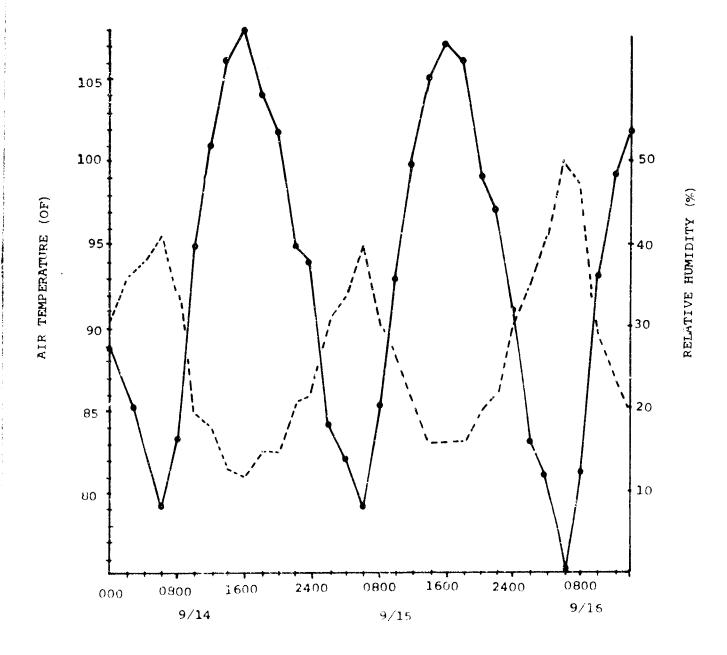


FIGURE 27
WEATHER CONDITIONS DURING FIELD TESTS
(WEATHER SQUADRON DATA, LUKE AFB, ARIZONA)

Observation of the pads from a distance disclosed an obvious difference in the appearance of the two surfaces.

The test aircraft made an average approach into P4d 1 (control pad) as testing began. Heavy dusting occurred, obscuring the aircraft completely from view in all quadrants around the area. A similar approach to Pad 2, however, created very slight dusting.

For the remainder of the day the test aircraft approached, landed and hovered over the surface of Pad 2. By 1600 hours (approximately 30 hours since pad preparation) dusting was becoming heavier but was not sufficient to present any significant problem according to observers from the sponsor. When testing was suspended for the day an additional three to five hours of operational time had been logged on the test pads.

16 September 1971 - Prior to arrival of the test aircraft, the areas surrounding the test pads were thoroughly soaked.

No water was added to the surfaces of either Pad 1 or Pad 2.

When testing commenced approximately 48 hours had elapsed since rad 2 had been treated with UPAC and dampened. As the test aircraft approached and landed relatively heavy dusting occurred, indicating that the effective period of the treatment had been surpassed. At that, however, the dusting created was not hearly as severe as that which occurred on the control pad. For that matter, it was not as severe as that which had occurred in the control area 24 hours before (9/15/71).

After operating over Pad 2 for approximately 30 minutes, testing was suspended and the pads retreated. The control

pad was thoroughly soaked with approximately 10,000 gallons of water. Pad 2 was dampened with approximately 300 gallons of water. After treatment of the pads they were allowed to "cure" for about two hours. Pad 2 was then subjected to heavy crisscross traffic with a Ferguson tractor.

The test aircraft then approached and landed on both test pads repeatedly. Low dust levels were observed in both areas. Close inspection of the surface of Pad 2 subsequent to repeated landings by the test aircraft disclosed that the soil and UPAC were loosely aggregated in a somewhat "tacky", fragile matrix. The presence of the swollen UPAC on the pad caused no problems in terms of slipperiness or poor footing (q.v., mud solidification below) nor did the soil-UPAC aggregate tend to stick excessively to vehicle wheels or the feet of individuals traversing the area.

Mud Solidification - Inconclusive results were achieved in this phase of testing due exclusively to difficulty in preparing a satisfactory "soft" mud with the test site soil.

After constructing an 18 inch high berm around Pad 3, the area was treated by inundating with 75,000 gallons of water (sufficient volume to produce a pond approximately 1.1 feet in depth). The water was applied between 1400 and 1600 hours, 13 September. Inspection of the site at 0800 hours on 14 September, showed however that there was no standing water on the pad and that the surface would easily support foot and vehicular traffic.

An additional quantity of 50,000 gallons was pumped onto the pad between 0830 and 1030 hours on 14 September, bringing the total depth of water applied to slightly over two feet. While water was still standing on the pad an area 15 feet square (located on the eastern perimeter of the pad) was then treated with 100 pounds of 10-30 mesh UPAC. The material was apread by hand by two men in approximately 15 minutes. No examt was made to mix the UPAC with the mud. The treated areas was left to "cure" for approximately rour hours. Inspection of the site at the end of that time revealed no standing water anywhere in the pad; the areas untreated with UPAC were sufficiently strong to support foot traffic. The treated area, however, being covered with a chick layer of swollen UPAC which lay on the firm earth surface wheath, was slippery and difficult to travers on foot because of that characteristic (q.v. dust suppression, above). The experiment was declared "no test" and further efforts in the area suspended.

Water Removal from POL. A 55-gallon drum was filled with a gallons of fuel oil from an underground reservoir. Samples of this POL were taken for analysis and labeled as controls. The POL was then contaminated with 1200 ml of distilled water, representing approximately one percent contamination of the fuel. The contaminated fuel was then agitated and samples taken to analyze for amount of contamination.

Two one-foot long cloth bags with an approximate diameter of 1 1/2 inches and each filled with ten grams of UPAC, we e used as water removal devices at the ottom of the drum. The cloth bags with UPAC were placed in the contaminated POL and allowed to sit for 40 hours. Results were as follows:

Control sample				88	ppm
Contaminated	sample			141	ppm
Contaminated	samples	after	treatment	111	ppm

Removal of the cloth bags show d that considerable water had been absorbed by the UPAC. The low efficiency of dehydration of the contaminated fuel was ascribed to poor agitation and a low level of contact between the UPAC and the water dissolved in the fuel.

<u>Disabling Vehicles</u>. Two junked cars were purchased for the field testing of UPAC in disabling vehicles. They were both equipped with tod Moto. Co. sign-cylinder engines with a cooling system capacity of 16 quarts. One car was used for cooling-system incapacitation while the other car was used for fuel-system incapacitation.

The cooling system incapacitation test was performed by adding 50 grams of 10-30 mesh UPAC to the radiator and waiting one hour before starting the engine. When the engine was started, it overheated in 14 minutes. A heater hose ruptured and the coolant water) boiled vigorously. Inspection of the engine after cooling showed that the radiator and radiator hoses were solidly plugged with UPAC.

The effort to incapacitate the fuel system of the second car was unsucconsful. Five grams of loose 10-60 mesh UPAC and 100 ml of water were added to the gas tank containing four gallons (15 gallon tank), and the system was allowed to "cure" for 16 hours. After that time the engine was started and ran at a fast idle for 30 minutes. There was no evidence of fuel starvation at any time. It is presumed that the water and UPAC settled in the fuel tank sump and did not enter any of the lines.

CONCLUSIONS AND RECOMMENDATIONS

The data acquired during the subject study establishes the feasibility for four military applications of UPAC. Each of these applications and the further work required to fully develop them is discussed separately below.

Water Removal from POL. POL materials purposely contaminated with water and then scrubbed by passage through a column of UPAC contain less moisture than the material did <u>before</u> contamination. Preliminary data suggests that flow through such columns can be in the order of several hundred gallons/minute. Other than dehydration, there appears to be no effect on POL products which have been in intimate contact with UPAC.

To develop this application further, chemical engineering studies are required to:

- (1) Determine parameters such as optimum column dimensions, maximum throughput volumes, hydraulic characteristics of the columns, column exhaustion and breakthrough characteristics, regeneration procedures, etc.
- (2) Perform detailed analyses of the POL materials treated with such columns to confirm that contact with the polymer has no effect on end use.
- (3) Design suitable prototype columns for test in the field on military equipment.

<u>Useful Work</u>. The expansion of UPAC as it absorbs water can be harnessed and made to move substantial weights. Although the generation of work energy is slow it appears to be wholly predictable with respect to time. Data acquired during the study indicates that temperatures above freezing will have little effect on the rate at which work is generated. These characteristics

suggest that UPAC may have application as the timing element in delays. In that role the work potential of the expanding material could be stored in a spring or similar device and released at the end of the delay. Such a unit need not rely on external power sources such as batteries, escapements, etc., either to perform the timing operation or the function desired at the end of the delay period.

To develop this application further study is required in order to:

- (1) Clearly identify the precision with which the delay increment can be measured.
- (2) Completely characterize the work function of the material as it is related to particle size, hydrostatic and mechanical pressure, temperature, etc.
- (3) Design prototype delay units and test those units in the field.

<u>Disabling Internal Combustion Engines</u>. Small quantities of UPAC introduced into the cooling system of a liquid cooled engine cause catastrophic failure of that system after 10-20 minutes of engine operation.

Preliminary data indicates that one to two grams of 30-60 mesh UPAC per quart of cooling capacity is sufficient to cause overheating. This ratio applies to coolants with ethylene glycol/water mixtures as high as 50/50. Inspection of engines disabled in this manner shows that a major effort is required to clear the stoppage and make the unit operational again.

Further development of this application will require study of:

- (1) Optimum quantities and particle size of UPAC.
- (?) Optimum packaging methods for introduction of the UPAC into cooling systems.

- (3) Effectiveness of the method in the field on engine types in use by the military.
- (4) Potential countermeasures.

<u>Dust Suppression</u>. Field tests show that it is possible to suppress dust during helicopter operations by treatment of the pad area with UPAC and a light water spray. As little as seven pounds of 10-60 mesh UPAC and 200 gallons of water per 1,000 square feet of surface is required. Exceptionally low dust conditions were achieved for as long as 30 hours on a pad treated in this way in spite of the adverse (high heat and low humidity) weather conditions during the test period. Dust control in the treated area was restored at the end of the test period by the application of a water spray of only 50 gallons per 1,000 square feet.

Further development of this technique will require:

- (1) Long term evaluation performed in the field under a variety of weather conditions and for prolonged periods of aircraft operation.
- (2) Systematic study to optimize treatment methods and material characteristics.